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EFFECTS OF INTERIM WARMING ON TENSILE PROPERTIES
OF A 286 STAINLESS STEEL
IRRADIATED AT CRYOGENIC TEMPERATURES

LOCKHEED NUCLEAR PRODUCTS
C. A. Schwanbeck, Project Manager

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-10298

LOCKHEED NUCLEAR PRODUCTS

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TOPICAL REPORT

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by

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C. A. Schwanbeck, Project Manager

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FOREWORD

This report is submitted to the National Aeronautics and Space Administration, Lewis Research Center, by the Lockheed-Georgia Company in accordance with the requirements of Article XXI, NASA Contract NAS 3-10298.

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LIST OF SYMBOLS

n/cm^2	fast fluence, in neutrons per square centimeter, with energies above 0.5 meV or 80 fJ
MeV	million electron volts
fJ	femtojoules
$^{\circ}\text{K}$	degrees Kelvin
$^{\circ}\text{R}$	degrees Rankine
Ksi	thousands of pounds (kips) per square inch
kN/cm^2	kilonewtons per square centimeter
MN/cm^2	meganewtons per square centimeter
F_{tu}	Ultimate tensile strength
F_{ty}	Tensile yield strength

1 SUMMARY

This report describes the results obtained in an experimental study of the effects of irradiation on the tensile properties of Type A-286 stainless steel, AMS 5735, at cryogenic temperatures. Test specimens were exposed to fluences of 6×10^{17} n/cm² and 9×10^{17} n/cm², with neutron energies greater than 0.5 MeV (80 femtojoules), at 17°K (30°R). Sets of specimens were irradiated continuously to the desired fluence; other sets were irradiated in increments of 3×10^{17} n/cm², with interim warming to 300°K (540°R), until the desired fluence was attained. The interim temperature was maintained for one hour (3.6×10^3 sec) between incremental irradiations. The specimens were tested in tension at 17°K (30°R) following the above environmental exposures. Other specimens were tested at 17°K (30°R) after irradiation to 3×10^{17} n/cm² followed by a post-irradiation annealing period of one hour (3.6×10^3 sec) at 300°K (540°R). Additional unirradiated control specimens were tested after being subjected to the thermal cycling received by the specimens which received interrupted irradiations.

The sample lots tested after continuous irradiation exposure showed no effect of irradiation on the ultimate tensile strength reliably discernable within the sensitivity of the test procedures. A small increase in the tensile yield strength was observed with increasing fluences; no such effect had been observed after exposure to 1×10^{17} n/cm² at 17°K (30°R) in an earlier program.

The effect of total irradiation fluence on the tensile yield strength appeared to be independent of interim warming during interrupted irradiations at the 95% confidence level (probability of obtaining a value equal to \pm the desired significance ratio is 0.05).

2 INTRODUCTION

The need for engineering evaluation of the combined neutron-irradiation cryogenic effects on structural materials has long been recognized by people responsible for advanced design in the aerospace program. Both low temperature environments and neutron bombardment produce embrittlement in many metals and alloys. Defects introduced by neutron irradiation are mobile even at the boiling point of liquid-hydrogen (20.5 degrees Kelvin, 36°R). Tests must, therefore, be conducted with the specimens held at the temperatures of interest during the entire irradiation and testing period, including thermal cycling to simulate environmental conditions anticipated in the structural members of a nuclear rocket.

In an earlier test program, authorized by Contract NASw-114 (ref. 1), Lockheed investigated the effect of fast neutron fluences of 10^{17} n/cm² ($E > 0.5$ MeV or 80 femtojoules) at 17°K (30°R) on the mechanical properties of a group of 33 structural alloys, including Type A-286 stainless steel. No statistically significant (at the 90% confidence level) adverse cryogenic or irradiation effects were observed in A-286 stainless steel in this program (ref. 1), and this alloy appeared suitable for further investigation as a possible material for nuclear rocket structures.

In deep space use the nuclear engine may shut down and re-start during flight. Therefore, it was deemed advisable to investigate the effects of interrupted cryogenic irradiation with interim warming to room temperature on Type A-286 stainless steel. The series of tests shown in Table I, authorized by Contract NAS 3-10298, were performed by Lockheed at the NASA Plum Brook Reactor Facility, to study these effects. A sample lot of three specimens was tested in tension after exposure to each of the environmental conditions shown in Table I.

The test results are reported and discussed in the following sections of this report.

3 TEST MATERIAL

The material under investigation in this test program was Type A-286 stainless steel. This material is a precipitation hardening austenitic steel sometimes classified as an iron-base superalloy. It is an excellent high-temperature alloy, with excellent resistance to corrosion in all atmospheres encountered in jet engine service at temperatures up to about 1300°F (980°K) coupled with good mechanical properties in the same temperature range (ref. 2). Type A-286 stainless steel has also shown good cryogenic properties in low temperature testing at various laboratories (ref. 3). Tests conducted at the Plum Brook Reactor Facility by Lockheed, in an earlier program showed no adverse effects due to irradiation to 10^{17} n/cm² at 17°K (30°R) (ref. 1).

A rather complex metallurgical structure is required to obtain the combination of desirable engineering properties mentioned above. The structure of A-286 stainless steel consists of a meta-stable austenitic matrix containing sub-microscopic precipitates of various intermetallic compounds produced by interaction between major and minor alloy constituents; these precipitates may have rather simple structures, such as Ni₄Mo or may be more complex as (xFe, yCr)₂Ti. Additional precipitate formation occurs through interaction of intentional alloy constituents with trace impurities. These also can be simple in form as Cr₄Sn or more complex, (xCr, yFe)₂₃C₆. The variety of combined forms which may possibly occur in an alloy of many diverse constituents is large and unpredictable. Therefore, this material is better suited for studies of engineering properties than for fundamental investigations for the formulation of theoretical models.

To avoid the introduction of an uncontrolled variable into the test program, all the test specimens were fabricated from a common lot of material; the same lot was used in the earlier program reported in reference 1. The material was melted by the Carpenter Steel Company using vacuum-consumable electrode techniques. The steel was cast into 20 inch (50.8 cm) square ingots and air cooled. The ingot was rolled to approximate size and heat treated as follows:

- . Hold at 1800°F (1253°K) for one hour
- . Quench in water
- . Hold at 1325°F (993°K) for 16 hours
- . Cool in still air.

This heat treatment conforms to the requirements of section 6.1.1 of AMS 5735 (ref. 4) and produces a Rockwell hardness of R_c 30-35.

The heat treated bars were centerless ground to a 0.50 inch (1.27 cm) diameter.

The chemical composition of this lot of material is shown in Table II.

4 TEST SPECIMENS

Due to space limitations in the irradiation access port (HB-2) and refrigeration capacity limitations, the specimen used was a miniaturization of the standard round specimen of ASTM E 8-66 (ref. 5), with a nominal gage diameter of 0.125 inch (0.318 cm) and a nominal gage length of 0.5 inch (1.27 cm). The tensile specimen used is shown in Figure 1. The ratios of the significant parameters are the same as for the standard ASTM specimen.

5 TEST EQUIPMENT AND PROCEDURES

The test procedures followed during this program were conducted, as nearly as feasible, in accordance with the provisions of The American Society of Testing and Materials Specifications ASTM E 184-62 and E 8-66 (ref. 5).

5.1 IRRADIATION TEST LOOP

All testing was performed at the NASA Plum Brook Reactor Facility using the horizontal beam port on the north face of the reactor core, designated as HB-2, as the irradiation facility. The testing machines are contained in cryogenic test loops capable of insertion into the 6" (15.24 cm) diameter beam port. Transfer tables provide the capability of insertion and withdrawal of the loops from HB-2 and provide rotation to permit positioning the loops in a radially aligned hot cave for specimen change. Specimen temperature control is maintained with an 1150 watt refrigerator using helium as the cryogenic fluid. Detailed descriptions of the test hardware may be found in references 1 and 6. The test loop is shown in Figure 2. The load control system, shown schematically in Figure 3, permits axial loading of the specimen in tension or compression with applied forces up to 5000 lbs (22,240 newtons).

for correcting this stress for the tri-axial state of stress during plastic instability are not available; therefore, this parameter as reported is of questionable reliability (ref. 7, p 246).

The accuracy and calibration of the test system are discussed in references 1 and 6.

6 TEST RESULTS

The results obtained during this test program, together with control data obtained in an earlier program (ref. 1), are given in Table III. Certain significant test parameters are plotted in Figure 4 for ease of comparison. A statistical analysis of the effects of test environment in the F_{tU} and F_{tV} values is given in Appendix A.

The basic purpose of this investigation, as stated in the introduction, was to observe the effects of interrupted cryogenic irradiation with interim warming periods to simulate the effects of cyclic start-up and shut-down of a nuclear rocket during a space mission. To permit assessment of these effects, a number of control specimens were tested. The entire program, for the basis of discussion, can reasonably be divided into three basic investigations:

- The investigation of the effects of cryogenic thermal cycling on unirradiated Type A-286 stainless steel;
- The investigation of the effects of cryogenic irradiation at several uninterrupted fluence levels on Type A-286 stainless steel;
- The investigation of the effects of interrupted cryogenic irradiation, with interim warm-ups, at several total fluence levels on Type A-286 stainless steel.

6.1 EFFECTS OF CRYOGENIC THERMAL CYCLING ON UNIRRADIATED TYPE A-286 STAINLESS STEEL

Austenitic and semi-austenitic steels consist, at least in a large degree, of a face centered cubic iron crystal lattice which is meta-stable at room temperature. In other words, the M_s (or temperature where the austenitic-martensitic transition begins) is only slightly above, or is actually below, room temperature. Sub-zero cooling may cause this transformation to occur, with large increases in strength parameters and reductions of ductility. Indeed, some semi-austenitic steels such as Type AM-350 stainless steel, have sub-zero exposures specified as part of the hardening cycle.

Examination of Figure 4 shows that the mean values from all F_{tU} data points fall within the range of values for the specimen lot tested at 17°K (30°R) without irradiation; no radiation effect is observable on the F_{tU} either for continuous or interrupted irradiation exposures.

6.3 EFFECT OF INTERRUPTED CRYOGENIC IRRADIATION WITH INTERIM WARMING

The mean values for the F_{ty} increase as a function of total fluence, as indicated by the dashed line connecting these values. There is no overlap in the range of values for the sample lot tested after continuous irradiation to 9×10^{17} n/cm² and that tested following three incremental irradiations of 3×10^{17} n/cm². However, the difference between these lots is not statistically significant at the 90% confidence level; if a difference actually exists it is too small for reliable determination using these experimental techniques.

A slight residual effect of irradiation to 3×10^{17} n/cm² at 17°K (30°R) is observable on the tensile yield strength of specimens tested at 17°K (30°R) following a post-irradiation anneal at 300°K (540°R).

Tests were not performed on specimens tested at 17°K (30°R) after exposures of 3×10^{17} n/cm² at 17°K (30°R) without annealing.

7 SUMMARY OF RESULTS

The following conclusions may be drawn from the data contained in this report:

1. There is no effect discernable, at the 90% confidence level, of interim warmings in interrupted irradiations on the absolute change in the tensile test parameter values after a constant total fluence up to $9 \times 10^{17} \text{ n/cm}^2$ ($E > 0.5 \text{ MeV}$). This does not preclude the possibility of such effects becoming evident after greater numbers of incremental exposures with interim warming nor of the presence of some effects on properties not determined by tensile testing.
2. There is no discernable effect of cryogenic cycling on the tensile properties of unirradiated Type A-286 stainless steel which might occur through austenitic-martensitic transformation at low temperatures.
3. There is no discernable effect of cryogenic-irradiation at 17°K (30°R) on the ultimate tensile strength or the ductility parameters of Type A-286 stainless steel at the fluence levels investigated (up to $9 \times 10^{17} \text{ n/cm}^2$).
4. There is a slight but discernable effect on the tensile yield strength from cryogenic-irradiation at 17°K (30°R) at a fluence of $6 \times 10^{17} \text{ n/cm}^2$, increasing at a fluence of $9 \times 10^{17} \text{ n/cm}^2$. This effect is not discernable after exposure to a total integrated flux of only $1 \times 10^{17} \text{ n/cm}^2$ at 17°K (30°R).
5. A slight residual effect of irradiation to $3 \times 10^{17} \text{ n/cm}^2$ at 17°K (30°R) is observable on the tensile yield strength of specimens tested at 17°K (30°R) following a post-irradiation anneal at 300°K (540°R).

8 REFERENCES

1. Lockheed Nuclear Products: Effect of Nuclear Radiation on Materials at Cryogenic Temperatures; Final Report, Contracts NASw-114 and NAS 3-7987, NASA CR-54881; LAC ER-8434, 1966.
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5. Anon.: ASTM Standards, The American Society for Testing and Materials, 1966.
6. Lockheed Nuclear Products: Effect of Nuclear Radiation on Materials at Cryogenic Temperatures; Final Report, Contract NAS 3-7985, NASA CR-72332; LAC ER-9757, 1967.
7. Dieter, George E. Jr.: Mechanical Metallurgy, McGraw-Hill Book Co., Inc., 1961.

9 TABLES

TABLE I SCOPE OF TEST PROGRAM

Test Class see Appendix A	Cyclic Test Conditions					Test Temp °K
	Irradiate at 17°K $\times 10^{-17}$ n/cm ²	Interim Temp, 1 hr °K	Irradiate at 17°K $\times 10^{-17}$ n/cm ²	Interim Temp, 1 hr °K	Irradiate at 17°K $\times 10^{-17}$ n/cm ²	
A *	unirradiated	-	-	-	-	300
B	unirradiated **	300	unirradiated **	300	-	300
C *	unirradiated **	-	-	-	-	17
D	unirradiated **	300	unirradiated **	-	-	17
E	unirradiated **	300	unirradiated **	300	-	17
F *	1	-	-	-	-	17
G	6	-	-	-	-	17
H	9	-	-	-	-	17
I	3	300	-	-	-	17
J	3	300	3	-	-	17
K	3	300	3	300	3	17

* Data obtained in earlier test program (ref. 1)

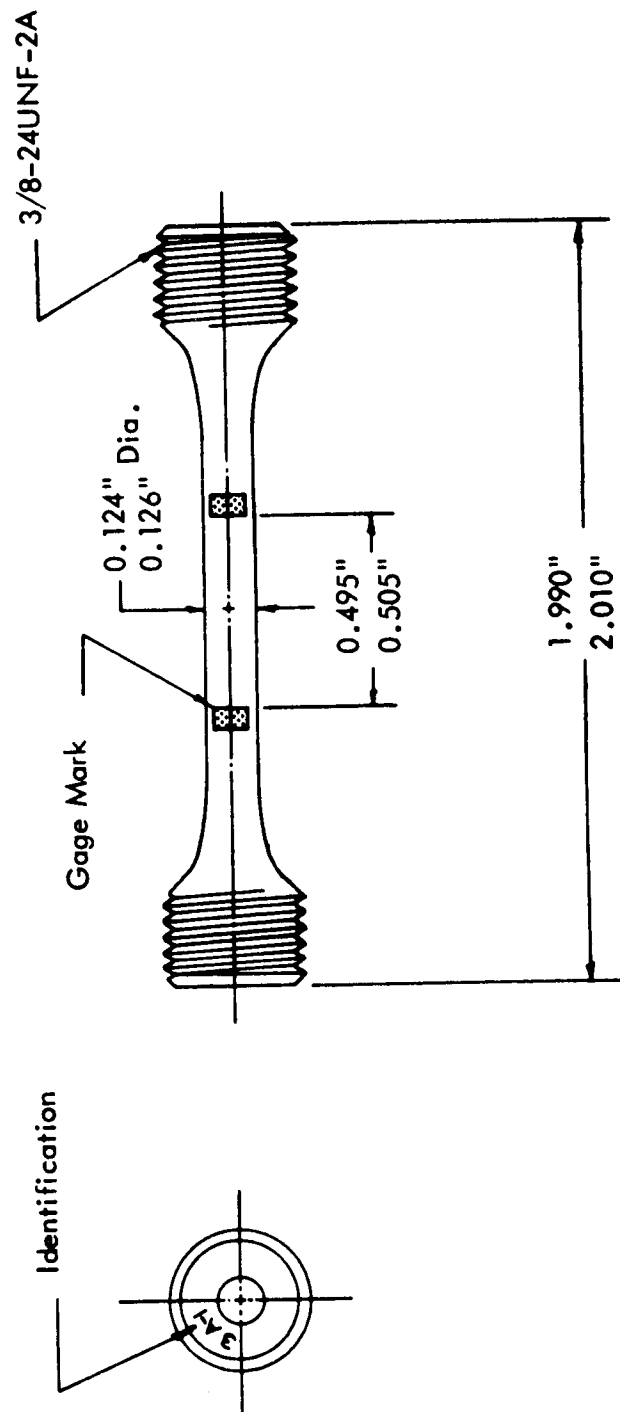
** Specimen stabilized at 17°K out-of-pile

TABLE III THE EFFECT OF IRRADIATION LEVEL AT 17°K, POST-IRRADIATION ANNEALING, AND INTERRUPTED IRRADIATION ON A-286 STAINLESS STEEL

Specimen	Irradiation at 17°K n/cm ²	Interim Temp. °K	Test Temp. °K	F _{tu} Ksi (kN/cm ²)	F _{ty} Ksi (kN/cm ²)	F _{ty} /F _{tu}	Elongation in 0.5 in (4D) %	Reduction of Area %	Fracture Stress Ksi (kN/cm ²)	Modulus E (#) 10 ³ Ksi (MN/cm ²)
Range of 5(a) Mean of 5(a)	None	---	300	153-158 156.0 (107.6)	110-116 112.2 (77.36)	0.70-0.74 0.720	25-27 26.3	49-55 51.0	(b) --- ---	28-31 (19-22) 30 (21)
6 Ca 30	[None]	300	---	156 (108)	113 (77.8)	0.72	26	51	250 (172)	29 (20)
6 Ca 58	[None]	300	300	161 (111)	112 (76.9)	0.69	26	50	263 (181)	32 (22)
6 Ca 61	[None]	300	300	162 (111)	117 (80.9)	0.73	26	51	268 (185)	29 (20)
Mean*				159.4 (109.9)	113.9 (78.51)	0.713	26.0	50.7	260.3 (179.5)	30 (21)
Range of 5(a) Mean of 5(a)	None	---	17	230-248 235.0 (162.0)	148-152 149.6 (103.2)	0.61-0.65 0.636	32-36 34.5	42-51 46.5	(b) --- ---	26-37 (18-26) 31 (22)
6 Ca 68	[None]	300	17	246 (169)	158 (109)	0.64	36	46	438 (302)	20 (14)
6 Ca 69	[None]	300	17	242 (170)	154 (106)	0.64	36	46	435 (300)	20 (14)
6 Ca 71	[None]	300	17	237 (163)	154 (106)	0.65	34	46	435 (300)	17 (12)
Mean*				241.5 (166.5)	155.3 (107.1)	0.643	35.3	46.0	436.0 (300.6)	19 (13)
6 Ca 63	[None]	300	---	234 (161)	151 (104)	0.65	36	46	416 (287)	30 (21)
6 Ca 64	[None]	300	17	236 (163)	151 (104)	0.64	35	45	431 (297)	34 (23)
6 Ca 77	[None]	300	17	231 (159)	146 (101)	0.63	35	44	415 (286)	30 (21)
Mean*				233.8 (161.2)	149.6 (103.1)	0.640	35.3	45.0	420.7 (290.1)	31 (21)
Range of 3(a) Mean of 3(a)	1 x 10 ¹⁷	---	17	228-230 229.0 (157.9)	149-157 152.0 (104.8)	0.65-0.68 0.663	33-34 33.5	14-18 16.0**	(b) --- ---	31-37 (22-26) 32 (22)
6 Ca 60	6 x 10 ¹⁷	---	17	238 (164)	165 (113)	0.69	33	46	430 (296)	36 (25)
6 Ca 75	6 x 10 ¹⁷	---	17	247 (171)	176 (122)	0.71	36	48	447 (308)	33 (23)
6 Ca 90	6 x 10 ¹⁷	---	17	247 (171)	163 (113)	0.67	35	46	451 (311)	30 (21)
Mean*				244.1 (168.3)	168.0 (115.9)	0.690	34.7	46.7	442.7 (305.2)	33 (23)
6 Ca 11	9 x 10 ¹⁷	---	17	247 (170)	180 (124)	0.73	27	36	388 (268)	35 (24)
6 Ca 56	9 x 10 ¹⁷	---	17	248 (171)	178 (123)	0.72	35	47	447 (308)	29 (20)
6 Ca 70	9 x 10 ¹⁷	---	17	240 (165)	175 (121)	0.73	33	46	433 (299)	32 (22)
Mean*				244.8 (168.8)	178.0 (122.7)	0.727	31.7	43.0	422.7 (291.4)	32 (22)
6 Ca 62	3 x 10 ¹⁷	300	17	238 (164)	160 (111)	0.67	34	46	424 (292)	32 (22)
6 Ca 80	3 x 10 ¹⁷	300	17	245 (169)	160 (111)	0.66	34	46	430 (296)	33 (23)
6 Ca 91	3 x 10 ¹⁷	300	17	241 (166)	158 (109)	0.66	34	46	428 (295)	32 (22)
Mean*				241.2 (166.3)	159.6 (110.1)	0.663	34.0	46.0	427.3 (294.6)	32 (22)
6 Ca 76	3 x 10 ¹⁷	300	---	240 (165)	164 (113)	0.69	33	46	416 (287)	34 (23)
6 Ca 93	3 x 10 ¹⁷	300	17	243 (168)	167 (115)	0.69	34	45	435 (300)	31 (21)
6 Ca 94	3 x 10 ¹⁷	300	17	243 (168)	163 (113)	0.67	34	46	435 (300)	31 (21)
Mean*				241.9 (166.8)	165.0 (113.8)	0.683	33.7	45.7	428.7 (295.6)	32 (22)
6 Ca 86	3 x 10 ¹⁷	300	---	245 (169)	174 (120)	0.71	32	46	443 (305)	33 (23)
6 Ca 87	3 x 10 ¹⁷	300	---	252 (174)	174 (120)	0.69	32	45	454 (313)	39 (27)
6 Ca 89	3 x 10 ¹⁷	300	17	242 (167)	167 (115)	0.69	32	49	471 (325)	32 (22)
Mean*				246.3 (169.8)	171.5 (118.2)	0.697	32.0	46.7	456.0 (314.4)	35 (24)

(a) From Table B 27, ref. 1. (b) Not Recorded
 ** These data are questionable in light of elongation data.
 # For Comparison Purposes Only
 * Calculated before rounding-off individual values

10 FIGURES



Note: Diameter at gage marks shall be center diameter + $0.002''$ - $0.004''$.

FIGURE 1 TENSILE SPECIMEN

APPENDIX A

SIGNIFICANCE OF THE DIFFERENCE BETWEEN TWO SAMPLE MEANS

To determine if the difference between the means of two groups of samples is statistically significant the null hypothesis, that the two sample means \bar{X}_1 and \bar{X}_2 are from the same population with respect to the population mean \bar{X}_p , is used. This hypothesis is tested by determining the probability of t , where t is the ratio of $\bar{X}_1 - \bar{X}_2$ to an estimate of the standard error of the difference between the two sample means.

The standard error of the difference between two sample means, $\sigma_{\bar{X}_1 - \bar{X}_2}$ is given by:

$$\sigma_{\bar{X}_1 - \bar{X}_2} = \sigma \sqrt{\frac{1}{N_1} + \frac{1}{N_2}} \quad (1)$$

where σ is the standard deviation of the population and N_1 and N_2 are the number of items in sample one and sample two, respectively.

Since the value of σ is unknown, its value must be estimated from the information given by the two samples. This estimate is $\hat{\sigma}_{1+2}$ obtained from:

$$\hat{\sigma}_{1+2} = \sqrt{\frac{\sum x_1^2 + \sum x_2^2}{N_1 - 1 + N_2 - 1}} \quad (2)$$

$\sum x_1^2$ and $\sum x_2^2$ can be obtained by:

$$\sum x^2 = \sum X^2 - \frac{(\sum X)^2}{N} \quad (3)$$

where X is the parameter value of each of the N items in the sample.

TABLE A-1

SUMMARY OF STATISTICAL ANALYSES OF SIGNIFICANCE
OF THE DIFFERENCE BETWEEN GROUP MEAN VALUES OF
STRENGTH DATA FROM A-286 STAINLESS STEEL, FOR
EFFECTS OF TEMPERATURE CYCLING WITHOUT
IRRADIATION

Statistical Parameters	Groups Compared		
ULTIMATE STRENGTH	A & B	C & D	D & E
$\hat{\sigma}_{1+2}$	2.47	6.94	3.65
$\hat{\sigma}_{\bar{X}_1 - \bar{X}_2}$	1.80	5.07	2.98
$\bar{X}_1 - \bar{X}_2$	3.4	6.5	7.7
t	1.89	1.28	2.58
P	0.10	0.25	0.06
Significant at P = .05	No	No	No
YIELD STRENGTH			
$\hat{\sigma}_{1+2}$	2.48	2.00	2.62
$\hat{\sigma}_{\bar{X}_1 - \bar{X}_2}$	1.81	1.46	2.14
$\bar{X}_1 - \bar{X}_2$	1.7	5.7	5.7
t	0.94	3.90	2.66
P	0.40	(a)	0.06
Significant at P = .05	No	Yes	No

(a) $0 < P < 0.01$

TABLE A-III

SUMMARY OF STATISTICAL ANALYSES OF SIGNIFICANCE
OF THE DIFFERENCE BETWEEN GROUP MEAN VALUES OF
STRENGTH DATA FROM A-286 STAINLESS STEEL, FOR
EFFECTS OF IRRADIATION AND TEMPERATURE CYCLING

Statistical Parameters	Groups Compared				
ULTIMATE STRENGTH	C & I	C & J	C & K	I & K	J & K
$\hat{\sigma}_{1+2}$	6.74	6.51	7.08	4.40	3.83
$\hat{\sigma}_{\bar{X}_1 - \bar{X}_2}$	4.92	4.75	5.17	3.57	3.11
$\bar{X}_1 - \bar{X}_2$	6.2	6.9	11.3	5.1	4.4
t	1.26	1.45	2.19	1.43	1.41
P	0.25	0.20	0.20	0.20	0.20
Significant at P = .05	No	No	No	No	No
YIELD STRENGTH					
$\hat{\sigma}_{1+2}$	1.63	1.91	2.76	2.97	3.22
$\hat{\sigma}_{\bar{X}_1 - \bar{X}_2}$	1.19	1.39	2.01	2.41	2.62
$\bar{X}_1 - \bar{X}_2$	10.0	15	22	11.9	6.5
t	8.40	11.1	10.9	4.94	2.48
P	(a)	(a)	(a)	(a)	0.07
Significant at P = .05	Yes	Yes	Yes	Yes	No

(a) $0 < P < 0.01$

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Abstract

Type A-286 Stainless Steel (AMS 5735) was tested in tension at 17°K (30°R) following irradiations of 3×10^{17} n/cm², 6×10^{17} n/cm² and 9×10^{17} n/cm². Parallel sets of specimens were irradiated continuously, at 17°K (30°R) to the total fluence and in incremental irradiation doses, at 17°K (30°R), of 3×10^{17} n/cm² with interim annealing periods of one hour (3.6×10^3 sec) at 300°K (540°R). No significant differences were noted in the mechanical properties measured after a given total fluence between specimens receiving continuous irradiations and those receiving incremental irradiations, with interim annealing, within the sensitivity of the experimental methods used. Yield strength values increased slightly with higher radiation levels.